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(54) Title: COLOR-CHANGING SYSTEMS FOR ORAL HYGIENE PRODUCTS

(57) Abstract

The present invention relates to color-changing systems for use in oral hygiene products. The color-changing systems in these products enable the user or a provider of dental services to determine when the oral hygiene product has been introduced into and retained within the oral cavity for a long enough time to assure that its desired oral hygiene function has been accomplished.

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COLOR-CHANGING SYSTEMS FOR ORAL HYGIENE PRODUCTS

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to color-changing systems for oral hygiene products. Oral hygiene products containing the color-changing systems of this invention are "self-indicating" as they enable the user or provider of dental services to readily determine when compliance with the manufacturer's suggested duration of use has been met. That is, the color-changing systems change the color of the oral hygiene product to indicate when the product has been retained within the oral cavity long enough to assure an effective oral hygiene treatment.

Discussion of Prior Art

Chemical compounds that provide or change color based upon pH changes in the environment to which they are exposed are well-known. Such compounds have been used in connection with products intended for use in the oral cavity. For instance, U.S. Patent No. 5,270,174 describes an indicator (such as methylene blue) that undergoes a characteristic color change under redox conditions as a means for detecting microbial activity in the mouth.

U.S. Patent No. 5,154,917 describes a mouth rinse that employs liquids of different colors contained in separate compartments within a multicompartment bottle. In this mouth rinse product, a therapeutically-

effective amount of an oral hygiene medicament is included in at least one of the different-colored liquids. The liquids are colored through the use of orally-acceptable indicator dyes, such as phenolphthalein, bromocresol purple, bromothymol blue, cresol red, phenol red and thymol blue. The liquids are maintained at pH levels that allow the color associated with that pH value to reach an aqua-colored solution at or near neutral pH when those liquids flow out of the bottle and are combined. The aqua-colored solution contains the oral hygiene medicament for introduction into the mouth.

International Patent Publication No. WO 89/07760 describes curcumin to be useful in the packaging materials of products intended for oral introduction for the purpose of detecting and warning of the presence of cyanide adulteration. In the presence of cyanide (which is highly alkaline), it is reported that curcumin will turn from its normal yellow color to an appearance of red to red-orange.

Japanese Patent Document JP 49-66,837 reports curcumin to be useful in dentifrice and mouthwash compositions to indicate effective cleaning by changing colors through adsorption phenomena in connection with a cellulose component in the compositions. Japanese Patent Document JP 49-71,151 reports xanthene to be useful in dentifrice and mouthwash compositions for that purpose.

To date, compounds that provide or change color based upon changes in pH values to indicate, through the color change, when an oral hygiene product has been retained within the oral cavity long enough to assure an effective oral hygiene treatment have not been suggested, formulated or used in such products.

Such a color-changing system would be desirable for oral hygiene products because it would indicate when the product had been retained within the oral cavity long enough to perform its desired oral hygiene function. A color-changing system of this nature would be desirable as an indicator to the user or provider of dental services of compliance with the manufacturer's suggested use requirements. The change of color of the product from an initial color to a completion color would indicate that the product had been retained within the oral cavity long enough to assure an effective oral hygiene treatment.

In addition, users of such color-changing system-containing oral hygiene products, particularly children, would benefit from such a color-changing system in the product as an educational aid to improve proper oral hygiene. There exists, therefore, a need for oral hygiene products containing color-changing systems.

SUMMARY OF THE INVENTION

The present invention relates to color-changing systems for oral hygiene products that "self-indicate" when the product has been introduced into and retained within the oral cavity long enough to achieve its desired oral hygiene function. That is, the change in color of the product is an indication that the oral hygiene treatment has been provided to the oral cavity long enough to be effective. Such a durational indicator should assist the user or provider of dental services in using the oral hygiene product in compliance with the manufacturer's recommendations. This durational indicator also aids users in achieving more effective oral hygiene.

This invention provides oral hygiene products, whose components include a color-changing system comprising color-changing materials and buffer systems, ingredients to provide oral hygiene treatments, and a vehicle base suitable to deliver the color-changing system and the oral hygiene ingredients into the oral cavity. In the color-changing systems used in this invention, the color-changing materials may be selected from a variety of materials that are capable of providing an initial color at a pH value of about 4 and a completion color, different from the initial color, at a pH value of about 7. Other color-changing materials may be selected from a variety of materials that are capable of providing an initial color at a pH value of about 8 and a completion color, different from the initial color, at a pH value of about 7.

Generally, it is undesirable to introduce materials into the oral cavity with a pH value greater than 8, because pH values more alkaline than 8 tend to irritate the oral cavity.

The color-changing materials employed in this invention neither impart a discernible taste or flavor to the oral hygiene product in which they are employed, nor are they intended to provide stain or color to the oral cavity itself. These color-changing materials are included in the oral hygiene products for the purpose of indicating when enough time has elapsed to assure an effective oral hygiene treatment.

This invention also provides a method for administering an oral hygiene product containing a color-changing system into an oral cavity, which product changes color when the product has been retained within the oral cavity long enough to assure an effective oral hygiene treatment.

In addition, this invention provides a method of using an oral hygiene product containing a color-changing system to indicate when the product has been introduced into and retained within an oral cavity long enough to assure an effective oral hygiene treatment.

DETAILED DESCRIPTION OF THE INVENTION

The oral hygiene products of this invention comprise (1) a color-changing system comprising color-changing materials and buffer systems, (2) ingredients to provide oral hygiene treatments and (3) a vehicle base suitable to deliver the color-changing system and the oral hygiene ingredients into the oral cavity. The oral hygiene products containing the color-changing systems of this invention allow the user or provider of dental services to determine when the oral hygiene product has been introduced into and retained within the oral cavity long enough to perform its desired oral hygiene function.

Color-Changing Systems

In the color-changing systems used in the present invention, the color-changing materials may be selected from a variety of materials that are capable of providing an initial color at a pH value of about 4 and a completion color, different from the initial color, at a pH value of about 7. Suitable materials include turmeric, carminic acid and derivatives thereof, red cabbage extract, anthocyanin-containing materials and anthocyanin derivatives.

The color additive turmeric is the ground rhizome of Curcuma longa L. See 21 C.F.R. § 73.600(a)(1). At a pH value within the range of from about 2.5 to about 6.5, turmeric exhibits a bright yellow color. At a pH

value above 6.5, the color changes to a completion color of orange.

The color additive carmine is the aluminum or calcium-aluminum lake of the coloring principle, chiefly carminic acid, obtained by aqueous extraction of cochineal [Dactolopius coccus costa (Coccus cactiL)]. The color additive cochineal extract is the concentrated solution obtained after removing the alcohol from an aqueous-alcoholic extract of cochineal [Dactylopius coccus costa (Coccus cactiL)]. The coloring principle is chiefly carminic acid. See 21 C.F.R. § 73.100(a)(1) and (2). Carmine is also referred to as CI Natural Red #4, carmine #40 and carmine #40 NF. For the purposes of this application, carmine, carminic acid and cochineal extract shall each be construed as referring to carminic acid.

At a pH value within the range of from about 3.5 to about 4, carminic acid exhibits an orange color. Above a pH value of about 5, carminic acid exhibits a blue-purple color. A preferred source of carminic acid includes cochineal extracts, such as No. 3401 cochineal extract and No. 3485 cochineal extract a.p., each of which is commercially available from Warner-Jenkinson.

Red cabbage extract exhibits a pink to red color at a pH value of about 3.8 or less. Above a pH value of about 4.5, red cabbage extract exhibits a blue color. A preferred source of red cabbage extract includes Nos. 3810 and 3815, each of which is commercially available from Warner-Jenkinson. The color-changing pigment of the red cabbage extract belongs to the family of flavanoids called anthocyanins. Other sources of anthocyanins or anthocyanin derivatives include extracts of berries like strawberry, raspberry and boysenberry; cherries and grapes; which extracts or anthocyanin-containing materials may be employed

herein. Of course, additional sources of anthocyanins and the anthocyanins themselves may be employed herein. For a more detailed discussion of anthocyanins, see G. Mazza and E. Miniati, Anthocyanins in Fruits, Vegetables, and Grains, CRC Press (1993), the disclosure of which is hereby incorporated herein by reference.

Other suitable color-changing materials include bromocresol green (yellow to blue), methyl red (red to yellow), bromocresol purple (yellow to purple) and chlorophenol red (yellow to red). Of course, combinations of these materials may also be employed. For a further discussion and other examples of such color-changing materials, see e.g., Daniel C. Harris, Quantitative Chemical Analysis, '241, W.H. Freeman & Co., San Francisco (1982), the disclosure of which is hereby incorporated herein by reference. Such color-changing materials described herein and others may be obtained commercially from, for instance, Warner-Jenkinson Company, Inc., a division of Universal Foods Corporation, St. Louis, Missouri, or Aldrich Chemical Company, Inc., St. Louis, Missouri.

Similarly, the color-changing materials may be selected from a host of materials capable of providing an initial color at a pH value of about 8 and a completion color different from initial color at a pH value of about 7. Compounds known to change color within this pH range of about 7 to about 8 include, but are not limited to, bromothymol blue (blue to yellow), phenol red (red to yellow), neutral red (orange to red), cresol red (red to yellow), α-naphthophthalene (blue to yellow) and cresol purple (purple to yellow). Of course, combinations of these materials may also be employed. These color-changing materials are widely available from commercial vendors, such as Aldrich.

These color-changing materials may be used in the color-changing systems in an amount effective to change the color of the color-changing system in which they are used when the pH value changes from an initial pH value to a completion pH value. In addition, the color imparted to the oral hygiene product by the color-changing material may be the same or different from an oral hygiene product formulated without the color-changing system as described herein.

Such an effective amount may vary depending on several factors, such as the chosen color-changing material, the effect of different components on the pH value of the particular formulation, the color intensity desired for a particular application and the length of time the oral hygiene product is to be retained within the oral cavity to provide oral hygiene treatments. Accordingly, persons skilled in the art may make appropriate choices as to types and amounts of color-changing materials that should be used depending on the particular application.

In the oral cavity, the type and amount of color-changing material chosen should be capable of providing a discernable difference in color between an initial color and a completion color of the color-changing system. This discernible difference in color of the oral hygiene product may be observed, for instance, within the oral cavity by the user or provider of dental services. Alternatively, the oral hygiene product may be expectorated from within the oral cavity after a sufficient duration, and the discernible color change observed, indicating that an effective oral hygiene treatment has been provided.

The color-changing materials suitable for use in this invention are sensitive to variations in pH values and exhibit one color at a certain pH value and another

color at another pH value. Thus, in a liquid delivery vehicle where an acidic pH value is desirable, the color-changing materials should be introduced into the oral cavity (where the physiological pH value is within the range of from about 6 to about 7) in a color-changing system contained in an oral hygiene product weakly buffered to a pH value below about 5. For instance, such a system may advantageously be included in a liquid delivery vehicle, such as LISTERINE® antiseptic mouth rinses (readily available commercially and manufactured by Warner-Lambert Company, Morris Plains, New Jersey), where after a duration within the range of from about 15 to about 45 seconds, preferably about 30 seconds, the color-changing system-containing mouth rinse changes color from an initial color at the more acidic pH value (e.g., about 4) to a completion color at a pH value of or closer to physiological pH (i.e., about 7).

In a liquid delivery vehicle where a more alkaline pH value is desired, such as in a fluoride mouth rinse, the color-changing system-containing fluoride mouth rinse changes color from an initial color at the alkaline pH value (e.g. about 8) to a completion color at a pH value of or closer to physiological pH (i.e., about 7). This color change may also occur after a duration within the range of from about 15 to about 45 seconds, preferably about 30 seconds, of use within the oral cavity. This color change, the duration of which may be varied by adjusting buffer capacities, indicates to the user or provider of dental services that the oral hygiene treatment has been effectively provided. When this duration has elapsed or the completion color has been reached, the user may expectorate the product from the oral cavity and be confident that an effective oral hygiene treatment has been obtained.

A buffer system should also be included in the color-changing systems to control the rate at which the color-changing materials change color when the oral hygiene products are introduced into and retained within the oral cavity -- i.e., the buffer allows the pH value to increase gradually during the time the oral hygiene product is retained within the oral cavity. By so controlling the rate of change of the pH value, the oral hygiene product may be retained within the oral cavity for a long enough time to allow the color-changing materials to change from their initial color to a completion color and assure an effective oral hygiene treatment.

Suitable acidic buffers include benzoic acid/benzoate, citric acid/citrate, acetic acid/acetate, succinic acid/succinate, phosphoric acid/phosphate and combinations thereof. The amount of buffer employed may vary depending on the particular oral hygiene product and the desired hygiene treatment to be administered to the oral cavity. A common acidic buffer is benzoic acid/benzoate, particularly sodium benzoate, in a ratio of about 0.025% (w/v) to about 0.018% (w/v). Suitable alkaline buffers include imidazole·HCl, N-2-hydroxyethyl piperazine-N'-2-ethanesulfonic acid ("HEPES"), phosphoric acid/phosphate and combinations thereof.

The oral hygiene product containing the color-changing system of this invention should be maintained at a pH value greater than about 4 to minimize demineralization of enamel. Within the oral cavity, the pH value increases to about 7 over the period of time the product is retained within the oral cavity due to salivary flow. That duration is typically within the range of from about 15 seconds to about 45 seconds, with about 30 seconds being preferred.

Alternatively, an oral hygiene product may be formulated with an alkaline pH value, preferably with a pH value of about 8, which during use would then change to the physiological pH of saliva. A duration similar to the acidic oral hygiene product above should elapse to attain the desired results.

Oral Hygiene Treatments

Any conventional oral hygiene treatment ingredient may be employed in the present invention, provided it is stable within the formulation and does not react adversely with any of the components of the formulations. Examples of such ingredients include plaque-loosening and/or plaque-removing ingredients; ingredients which retard plaque growth; fluoride-releasing ingredients to fight tooth decay; ingredients to prevent or minimize gingivitis; breath freshening ingredients; bacteriocidal and bacteriostatic agents to prevent or minimize microbial infection in the oral cavity; desensitizing agents; tartar-control agents and combinations thereof.

Plaque-loosening ingredients and/or plaque removing ingredients suitable for use herein as an oral hygiene treatment include abrasives such as dicalcium phosphate, silicon dioxide, calcium carbonate, aluminosilicates, sodium bicarbonate and the like, and combinations thereof.

Plaque-retarding ingredients suitable for use herein as an oral hygiene treatment include the combination of thymol, menthol, eucalyptol and methyl salicylate (the active essential oil ingredients in LISTERINE® antiseptic mouth rinses), quaternary ammonium compounds like alkyl pyridinium halides (e.g., cetyl pyridinium chloride, benzethonium chloride and benzalkonium

chloride and the like), triclosan (2,4,4'-trichloro-2-hydroxydiphenyl ether) and combinations thereof.

Fluoride-releasing ingredients are compounds characterized by their ability to release fluoride ions or fluoride-containing ions. Due to chemical instability of many fluoride-releasing compounds at acid pH values, these compounds are generally used in alkaline delivery vehicles to provide their intended treatment. Suitable fluoride-releasing compounds for use herein as an oral hygiene treatment include inorganic fluoride salts, such as water-soluble alkali metal salts, alkaline earth metal salts and heavy metal salts, such as sodium fluoride, potassium fluoride, stannic fluoride, stannous fluoride, barium fluoride, sodium fluorosilicate, ammonium fluorosilicate, sodium fluorozirconate, sodium monofluorophosphate, aluminum mono- and di-fluorophosphates, fluorinated sodium calcium pyrophosphate and combinations thereof.

Anti-gingivitis ingredients suitable for use herein as an oral hygiene treatment include the combination of thymol, menthol, eucalyptol and methyl salicylate (the active essential oil ingredients in LISTERINE® antiseptic mouth rinses), and bisbiguanides (like chlorhexidine).

Breath fresheners suitable for use herein as an oral hygiene treatment include zinc salts (like zinc chloride, zinc gluconate, zinc salicylate and the like), copper salts (like copper gluconate and the like) and combinations thereof.

Bacteriocidal and bacteriostatic agents suitable for use herein as an oral hygiene treatment include the combination of thymol, menthol, eucalyptol and methyl salicylate (the active essential oil ingredients in LISTERINE® antiseptic mouth rinses), quaternary

ammonium compounds like those noted above, bisbiguanides like those noted above, triclosan and combinations thereof.

Desensitizing ingredients suitable for use herein as an oral hygiene treatment include stannous fluoride, citric acid and salts (e.g., sodium) thereof, potassium nitrate, strontium chloride, calcium phosphate and combinations thereof.

Tartar-control agents suitable for use herein as an oral hygiene treatment include stannous fluoride, pyrophosphates, such as sodium, calcium or potassium pyrophosphate, chelating agents, such as ethylenediaminetetraacetic acid (EDTA) and citric acid, and combinations thereof.

Hydrogen peroxide and/or sodium bicarbonate may also be included in the oral hygiene products containing the color-changing system of this invention to provide an oral hygiene treatment.

Administration of Oral Hygiene Products Containing Color-Changing Systems

Like many over-the-counter products, oral hygiene products containing the color-changing systems of the present invention may be introduced into the oral cavity through a variety of delivery vehicles. Suitable delivery vehicles for these products include solid delivery vehicles, liquid delivery vehicles and semi-solid delivery vehicles. Suitable solid delivery vehicles include lozenges, chewing gums, tablets, powders, and the like; suitable liquid delivery vehicles include mouthwashes, rinses and sprays and the like; and suitable semi-solid delivery vehicles include suspensions, balms and dentifrices such as toothpastes, dental gels and the like.

In accordance with this invention, an effective amount of a color-changing compound may be admixed with an appropriate buffer system to form a color-changing system, which may then be added to a suitable delivery vehicle base. The effective amount of the color-changing compound may be readily determined by those skilled in the art without the need for undue experimentation. The effective amount may vary depending on the particular compound or combination of compounds chosen. For instance, a color-changing liquid system may be prepared by adding a color-changing material in an amount within the range of from about 0.01% to about 1% and a buffer system, such as benzoic acid/benzoate, and thereafter adjusting the pH value of this color-changing system level to about 4. This color-changing system may then be added to a delivery vehicle base, to which was previously added an oral hygiene treatment ingredient as described herein, to bring the total amount of all components of the oral hygiene product to 100% by weight.

Liquid Delivery Vehicles

Liquid delivery vehicles, such as mouthwashes, rinses, sprays and the like, may be used to introduce and deliver the color-changing system into the oral cavity.

When delivery in the form of a liquid is desirable, the oral hygiene product may comprise an effective amount of a color-changing system (comprising a color-changing compound with an appropriate amount of a buffer system to indicate when an effective amount of an oral hygiene treatment has accomplished its intended function within the oral cavity) and an active oral hygiene ingredient, mixed together with a liquid base to form a solution, suspension or dispersion. For example, oral hygiene products in liquid form may be prepared from about 0.02% to about 20% of a color-changing system, about

0.01% to about 30% of an ingredient to provide an oral hygiene treatment and about 50% to about 99.07% of the liquid base.

In the liquid form, a color-changing system may be prepared, for instance, by first dissolving in water a color-changing compound with a suitable buffer system. Then sufficient water, orally-acceptable solvents, surfactants, syrups or combinations thereof, may be added as the liquid base with mixing until reaching the desired volume. Ingredients for oral hygiene treatments, such as those described herein, may then be included at this stage of preparation.

The oral hygiene product containing a color-changing system of this invention may also be in the form of a suspension, which may be prepared by conventional methods. Suspensions may also contain those materials typically employed in formulating suspensions known in the art. For instance, the suspensions may be prepared by admixing the thickener with water, and heating the mixture to a temperature within the range of from about 40°C to about 70°C. If the thickener is not water-soluble a dispersion may form and if the thickener is water-soluble a solution may form. Oral hygiene treatments, such as those described herein, may then be admixed with water. The color-changing system may then be admixed with the thickener-water mixture. The oral hygiene treatment-containing thickened mixture and the color-changing system-containing thickened mixture may then be mixed together with agitation until substantial uniformity is reached.

Semi-Solid Delivery Vehicles

Dentifrices like toothpastes or dental gels, balms, ointments, salves, creams and suspensions are typically included when speaking of semi-solid delivery vehicles.

While the present invention is appropriate for semi-solid delivery vehicles generally, for reasons of brevity, toothpastes and dental gels are further described herein.

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Toothpastes and Dental Gels

When delivery in the form of a dentifrice like a paste or gel is desirable, the oral hygiene product should
10 comprise a color-changing system, an ingredient to provide an oral hygiene treatment, mixed together with a toothpaste base or a dental gel base.

In such dentifrice compositions, the toothpaste or
15 dental gel delivery vehicle base generally comprises water, typically in an amount within the range of from about 20% to about 40% by weight. Polyethylene glycol, polypropylene glycol, glycerin and combinations thereof may also be present in the delivery vehicle to serve as
20 humectants or binders in amounts within the range of from about 20% to about 60% by weight. The base may often include gelling agents or thickening agents, such as natural or synthetic gums or gelatin like hydroxyethyl cellulose, cellulose, methyl cellulose,
25 glycerin, carboxypolymethylene, and the like, and combinations thereof. Gelling agents or thickening agents may be used in an amount within the range from about 0.1% to about 25% by weight.

30 The toothpaste or dental gel compositions of the present invention may contain conventional additives, such as polishing agents, abrasive agents, sweeteners, flavoring agents and the like, provided these additives are inert to the color-changing compounds of the color-
35 changing system.

In paste form, calcium carbonate or calcium dihydrate may be used as a polishing agent. In gel form,

colloidal silica and/or alkali metal aluminosilicate complexes may be employed as polishing agents since these materials have refractive indices similar to the refractive indices of the gelling systems commonly used in dental gels. These polishing agents may be used in an amount up to about 25% by weight of the toothpaste or dental gel composition.

The present invention extends to methods for preparing such toothpaste or dental gel compositions. In these methods, the toothpaste or dental gel compositions may be prepared by admixing an effective amount of a color-changing system (i.e., color-changing compound with an appropriate buffer system), an effective amount of an ingredient to provide an oral hygiene treatment and an appropriate amount of a paste or gel delivery vehicle base. The oral hygiene product may be readily prepared using conventional methods and apparatus.

For instance, oral hygiene products in the form of toothpastes or dental gels may be prepared by dispersing a gelling agent in a humectant, water or combinations thereof. The resulting dispersion should then be admixed with an aqueous solution of an ingredient to provide an oral hygiene treatment and additives conventionally included in such dentifrices. A polishing agent and a flavoring agent may then be admixed therewith. Finally, color-changing systems of this invention may then be admixed therewith. The prepared toothpaste or dental gel composition may then be tubed or otherwise packaged. The liquid components and solid components in such a product should be proportioned to form a creamy or gelled mass that may be extruded from a pressurized container, from a collapsible tube or from other suitable containing dispensers, such as a pump, though not limited to those pumps now commercially available.

Solid Delivery Vehicles

In the context of solid delivery vehicles, as noted above, lozenges, chewing gums, tablets, powders and the like are typically included.

Lozenges

Lozenges are intended to be convenient, portable solid dosage forms. Lozenges may be produced in a variety of shapes such as flat, circular, octagonal and biconvex forms. When delivery in the form of lozenges is desirable, the oral hygiene product may comprise a color-changing system (i.e., color-changing compound with suitable buffer) and an ingredient to provide an oral hygiene treatment, mixed together with a lozenge base.

Lozenge bases are generally in two forms: hard boiled candy lozenges and compressed tablet lozenges. Hard boiled candy lozenges may be processed and formulated by conventional means, such as those involving fire cookers, scraped surface cookers and vacuum cookers. In general, a hard boiled candy lozenge has a base composed of a mixture of sugar and other carbohydrate bulking agents kept in an amorphous or glassy condition. This amorphous or glassy form is considered a solid syrup of sugars, generally having a moisture content of from about 0.5% to about 1.5%. Such materials normally contain up to about 92% syrup, up to about 55% sugar and from about 0.1% to about 5% water by weight of the final composition. The syrup component is generally prepared from corn syrups, such as those having a high fructose content.

Boiled candy lozenges may also be prepared from non-fermentable sugars, such as sorbitol, mannitol and hydrogenated corn syrup, and include LYCASIN

(commercially available from Roquett Corporation) and HYSTAR (commercially available from Lonza, Inc.). Candy lozenges may contain up to about 95% sorbitol, a mixture of sorbitol and mannitol in a ratio within the range of from about 9.5:0.5 to about 7.5:2.5 and hydrogenated corn syrup up to about 55% by weight of the solid syrup component.

Fire cookers involve the traditional method of making a boiled candy lozenge base. In this method, the desired quantity of carbohydrate bulking agent should be added to water and heated in a kettle until it dissolves. Additional bulking agent may then by added, if desired, with cooking continued until a final temperature within the range of from about 145°C to about 156°C is achieved. The batch may then be cooled and worked as a plastic-like mass, with any additional ingredients incorporated therein at this stage.

Scraped surface cookers (or high-speed atmosphere cookers) use a heat-exchanger surface which involves spreading a film of candy on a heat exchange surface. The candy may then be heated quickly to a temperature within the range of from about 165°C to about 170°C. The candy may then be rapidly cooled to a temperature within the range of from about 100°C to 120°C and worked as a plastic-like mass, with any additives, such as those described herein, incorporated at this stage into the plastic-like mass.

In vacuum cookers, the carbohydrate bulking agent is boiled to a temperature within the range of from about 125°C to about 132°C, with vacuum being applied and additional water boiled off without extra heating. When cooking is complete, the mass appears as a semi-solid and possesses a plastic-like consistency. At this stage, ingredients for oral hygiene treatments

such as those described herein, may be admixed into the mass by routine mechanical mixing operations.

Once the boiled candy lozenge has been properly tempered, it may be cut into workable portions or formed into desired shapes. A variety of forming techniques may be utilized depending upon the shape and size of the final product desired.

Compressed tablet lozenges often contain sugars as bulking agents in an amount up to about 95% by weight of the composition. Tablet excipients like binders and lubricants as well as sweeteners, flavoring agents, coloring agents and the like may also be added. For particular examples of such excipients and other additives, see infra.

Oral hygiene products containing the color-changing system of this invention in the form of lozenges may be made of soft confectionery materials. Conventionally, soft confectionery, such as nougat, toffees, chewy candies and the like, are prepared by combining two primary components: (1) a high boiling syrup, such as a corn syrup, hydrogenated starch hydrolysate and the like, and (2) a relatively light textured frappe, generally prepared from egg albumin, gelatin, vegetable proteins, such as soy derived compounds, sugarless milk-derived compounds, such as milk proteins, and combinations thereof. The frappe is generally relatively light, and may have a density within the range of from about 0.5 to about 0.7 grams/cc.

The high boiling syrup ("bob syrup") of soft confectionery materials is relatively viscous and has a density higher than the frappe component, and frequently contains a substantial amount of carbohydrate bulking agent, such as a hydrogenated starch hydrolysate. Conventionally, the final nougat

composition is prepared by the addition of the bob syrup to the frappe under agitation, to form the basic nougat mixture. Ingredients for oral hygiene treatments, such as those described herein, may be added thereafter also under agitation. A general discussion of nougat confection and its preparation may be found in B.W. Minifie, Chocolate, Cocoa and Confectionery: Science and Technology, 424-25, 3rd ed., Van Nostrand Reinhold, New York (1989), the disclosure of which is hereby incorporated herein by reference.

In general, the frappe component may be prepared first and thereafter the syrup component slowly added under agitation at a temperature of at least about 65°C, with a temperature of at least about 100°C being desirable. Mixing should be continued until substantial uniformity is reached, at which time the mixture should be cooled to a temperature below about 80°C, and additives and/or ingredients for oral hygiene treatments, such as those described hereinafter, may be added, if desired. Mixing may be continued for an additional period of time until the mixture is ready to be removed and formed into suitable confectionery shapes.

Chewing Gum

When delivery in the form of a chewing gum is desirable, the oral hygiene product may comprise a color-changing system and an ingredient to provide an oral hygiene treatment, mixed together with a gum base.

The gum base may be any conventional water-insoluble gum base, which includes those gum bases utilized for chewing gums and bubble gums. Illustrative examples include, without limitation, natural and synthetic polymeric materials like elastomers and rubbers. For example, polymeric materials suitable as gum bases include substances of vegetable origin such as chicle,

crown gum, nispero, rosadinha, jelutong, perillo, niger gutta, tunu, balata, gutta-percha, lechi-capsi, sorva, gutta kay and the like; synthetic elastomers such as butadiene-styrene copolymers, polyisobutylene, isobutylene-isoprene copolymers, polyethylene and the like; and combinations thereof.

The amount of polymeric material employed in the gum base may vary considerably depending upon various factors such as the type of gum base used, the consistency of the gum base desired, and the other components used to make the final chewing gum product. In general, the polymeric material is present in the gum base in an amount within the range of from about 5% to about 50%, preferably from about 15% to about 25%, by weight based on the total weight of the gum base.

Bulking agents suitable for use in gum bases include monosaccharides, disaccharides, polysaccharides, sugar alcohols, polydextrose, maltodextrins, mineral adjuvants, which serve as fillers and textural agents (such as calcium carbonate, talc, titanium dioxide, dicalcium phosphate and the like) and combinations thereof. Bulking agents may be used in the gum base in an amount up to about 90% by weight of the final chewing gum composition, with an amount within the range of from about 40% to about 70% by weight being desirable and about 50% to about 65% being more desirable.

The gum base may also contain a variety of traditional ingredients desirable to modify the texture and/or consistency or other properties of the final chewing gum product. These ingredients include plasticizers or softeners like lanolin, palmitic acid, oleic acid, stearic acid, sodium stearate, potassium stearate, glyceryl triacetate, glyceryl lecithin, glyceryl monostearate, propylene glycol monostearate, acetylated

monoglyceride, glycerin, polyethylene glycol, glycerol, sorbitol, dioctyl-sodium sulfosuccinate, triethyl citrate, tributyl citrate, 1,2-propyleneglycol, mono-, di-, tri-acetates of glycerol and the like, and combinations thereof. In addition, waxes (e.g., natural and synthetic waxes like petroleum waxes, such as polyurethane waxes, polyethylene waxes, paraffin waxes, microcrystalline waxes and fatty waxes), vegetable oils (e.g., coconut oil, palm kernel oil and the like) sorbitan monostearate, animal fats (e.g., tallow and the like), propylene glycol and the like may also be desirable to modify the texture and/or consistency or other properties of the final chewing gum product. Such plasticizers may be used in an amount of up to about 25%, and preferably in an amount within the range of from about 1% to about 17%, by weight of the gum base.

The present invention extends to methods of making the chewing gum oral hygiene products. The color-changing system of the inventor and the ingredients for oral hygiene treatments may be incorporated into an otherwise conventional chewing gum using standard techniques and equipment known to those skilled in the art.

Chewing gum bases may be prepared by batch methods. Such methods generally involve mixing and melting the components of the gum base in kettle mixers in numerous stages, placing the resulting homogenous mass on trays to be cooled, dried and thereafter transferred for incorporation into a chewing gum.

Chewing gum bases may also be prepared by continuous processes, such as with a twin screw extruder. See U.S. Patent Nos. 4,555,407; 5,045,325; and 5,135,760, the disclosures of each of which are hereby incorporated herein by reference.

For example, an appropriate chewing gum base may be chosen and heated to a temperature sufficiently high to soften the base without adversely effecting the physical and chemical properties and characteristics of the base. While the optimum temperatures may vary depending upon the composition of the chewing gum base, such temperatures may be readily determined by those skilled in the art without undue experimentation.

Conventionally, the gum base may be heated at a temperature within the range of from about 60°C to about 120°C for a period of time sufficient to soften and render it molten. For example, the chewing gum base may be heated under these conditions for a period of time of about thirty minutes just prior to being admixed incrementally with the remaining ingredients of the base. The chewing gum base may then be blended with the color-changing system and the ingredient for oral hygiene treatments may have been previously blended with ingredients for oral hygiene treatments. Mixing may be continued until a substantially uniform chewing gum composition is obtained. Thereafter, the chewing gum composition may be formed into shapes desirable for chewing gum.

The chewing gum composition may be prepared with a liquid-filled center, which contains the color-changing system and ingredients for oral hygiene treatments. Alternatively, the liquid-filled center of the chewing gum may contain one of the color-changing systems or the ingredients to provide oral hygiene treatments, and the other contained in the chewing gum base itself, or vice versa.

Tablets

When delivery in the form of tablets is desirable, the oral hygiene product may comprise a color-changing

system and an ingredient to provide an oral hygiene treatment mixed together with a suitable amount of a tablet base.

The tablet base may comprise a bulking agent which may be selected from a wide variety of materials such as dextrose, lactose, sugar, corn syrup and combinations thereof, and in the case of sugarless bulking agents, sugar alcohols such as sorbitol, mannitol and combinations thereof.

Tablets may be prepared in a variety of shapes (e.g., round, oval, oblong, cylindrical and triangular), and are available in two classes -- compressed tablets and molded tablets. For a more detailed discussion, see Remington's Pharmaceutical Sciences, chap. 89, 1633-59, 18th ed. (1990), the disclosure of which is hereby incorporated by reference.

Tablet preparation is well known. For a comprehensive discussion on that topic as well as on powder formulations, see Remington's, chap. 88, 1625-32.

Additives

In any of the forms of delivery vehicle in which the oral hygiene products containing color-changing systems may be prepared, certain additives may also be included. Examples of such additives include orally-acceptable solvents, sweeteners, flavoring agents, anti-foaming agents, humectants, lubricants, disintegrating agents, dyes or coloring agents, preservatives or shelf-life enhancing agents, organoleptic consistency modifiers or bioadhesives (like suspending agents, gelling or thickening agents) and combinations thereof, provided the additives are inert to the color-changing systems and the ingredients to provide oral hygiene treatments.

Orally-acceptable solvents such as ethyl alcohol, propylene glycol, polyethylene glycol and the like may be used to dissolve the flavoring agents and the active oral hygiene ingredients. In general, orally-acceptable solvents may be used in an amount up to about 30% by weight, with about 2% to about 5% being desirable. Orally-acceptable solvents may be employed to aid in dissolving, suspending or dispersing any one or more ingredients of the color-changing system and/or the oral hygiene product containing the color-changing system of this invention during preparation thereof.

Surfactants may also be used herein to act as solubilizers. Surfactants desirably reduce surface tension when dissolved in water or reduce interfacial tension between two liquid components or a liquid component and a solid component. Suitable surfactants include compounds which are capable of solubilizing the color-changing compounds and/or the active oral hygiene ingredients and which are inert thereto. The surfactants may be nonionic surfactants, anionic surfactants, cationic surfactants, amphoteric surfactants and combinations thereof. Suitable surfactants may be found listed and described in McCutcheon's Emulsifiers and Detergents, North American ed. (1988).

It may be desirable to use sweetening agents (sweeteners) in any of the forms of delivery vehicles in which the oral hygiene product containing the color-changing system according to this invention may be prepared. While certain sweeteners may be more desirable when used in one delivery form than another, such sweeteners well known in the art, including both natural and alternative sweeteners, may be employed herein with one of ordinary skill in the art making appropriate choices of among those sweeteners. The sweeteners suitable for use herein may be selected from

water-soluble sweeteners, water-soluble alternative sweeteners, water-soluble sweeteners derived from naturally-occurring water-soluble sweeteners, dipeptide-based sweeteners, protein-based sweeteners, sugar alcohols and combinations thereof.

Without being limited to particular sweeteners, representative classes and examples of such include:

(a) water-soluble sweeteners such as monosaccharides, disaccharides and polysaccharides [like xylose, ribose, glucose (dextrose), mannose, galactose, fructose (levulose), sucrose (sugar) and maltose], invert sugar (a mixture of fructose and glucose derived from sucrose), partially hydrolyzed starch, corn syrup solids, dihydrochalcones, monellin, steviosides, glycyrrhizin and combinations thereof;

(b) water-soluble alternative sweeteners, such as saccharin and salts (e.g., sodium, calcium or potassium) thereof, cyclamate salts (e.g., sodium or calcium cyclamate salts), sodium, ammonium or calcium salts of 3,4-dihydro-6-methyl-1-2,3-oxathiazin-4-one-2,2-dioxide, potassium salt of 3,4-dihydro-6-methyl-1,2,3-oxathiazin-4-one-2,2-dioxide (known under the designation ACESULFAME-K) and combinations thereof;

(c) dipeptide-based sweeteners such as L-aspartic acid derived alternative sweeteners, such as α-L-aspartyl-L-phenylalanine methyl ester (commercially available from the Nutrasweet Company under the trademark ASPARTAME®), α-L-aspartyl-N-(2,2,4,4-tetramethyl-3-thietanyl)-D-alanin-amide hydrate (known under the designation ALITAME), methyl esters of α-L-aspartyl-L-phenylglycine and α-L-aspartyl-L-2,5-dihydrophenyl-glycine, α-L-aspartyl-2,5-dihydro-L-phenylalanine; α-L-aspartyl-L-(1-cyclohexen)-alanine and combinations thereof;

(d) water-soluble sweeteners derived from naturally occurring water-soluble sweeteners, such as chlorinated sucrose derivatives -- e.g., chlorodeoxysucrose or chlorodeoxygalactosucrose (known under the designation SUCRALOSE);

(e) protein-based sweeteners, such as thaumaoccous danielli (Thaumatin I and II); and

(f) sugar alcohols, such as sorbitol, xylitol, inositol, maltitol, mannitol and combinations thereof.

Of course, combinations of sweeteners across these classifications may also be used.

A sufficient amount of sweetener should be used to provide the level of sweetness desired in the particular composition. The amount of sweetener chosen may be within the range from about 0.0025% to about 90% by weight, depending of course on the particular sweetener, its physical properties and characteristics and the level of sweetness desired.

Flavoring agents may also be used herein, including natural flavors and natural flavor mimetics. Indeed, any flavoring agent generally recognized as safe for food and drug application may be used herein. Suitable flavoring agents include mints (such as peppermint and spearmint), citrus flavors (such as orange and lemon), vanilla, cinnamon, various fruit flavors (such as cherry and apple) and the like. The amount of flavoring agent may vary depending of course on the specific flavoring agent, its physical properties or characteristics and the intensity of the flavor imparted. It is noteworthy that flavoring agents may affect the pH values of the formulations in which they are employed. Therefore, buffer systems should be adjusted accordingly.

Typically, the amount of sweetener and/or flavoring agent employed in the composition is a matter of preference subject to such factors as the type of composition and delivery vehicle thereof, the rapidity of delivery of the particular sweetener and/or flavoring agent employed and the intensity desired.

Anti-foaming agents, such as dimethyl polysiloxane, may be advantageously used herein. When used, the anti-foaming agent should be employed in an amount up to about 0.2% by weight, with about 0.01% to about 0.1% being desirable.

Suitable humectants useful herein include glycerin, sorbitol, mono- and di-glycerides of fatty acids, propylene glycol, pectins and the like, and combinations thereof. When used, humectants may be employed in an amount within the range of from about 1% to about 35% by weight.

Suitable lubricants include lipids (like oils and fats), euricamide, waxes and phospholipids (like unsaturated and saturated fatty acids and salts thereof, such as aluminum, calcium, magnesium and tin stearates), silicones and the like may be used in an amount within the range of from about 0.001 to about 10% by weight.

Suitable disintegrants or disintegrating agents for solid delivery vehicles, like tablets, include starches, cellulose gums and combinations thereof.

Suitable dyes, opaquing agents or coloring agents may be advantageously employed herein to complement the color-changing capabilities of the color-changing system. When used, the dyes or opaquing agents include titanium dioxide, silicon dioxide, coloring agents, such as azo-dyes, and other dyestuffs and pigments,

such as iron oxides, titanium dioxides, natural dyes and the like may be used in an amount within the range of from about 0.001 to about 10% based upon the weight of the composition.

Preservatives or shelf-preservation agents, such as butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), parabens like methyl paraben and propyl paraben, tocopherols and the like, and combinations thereof may be employed herein. As with flavoring agents, these preservatives may affect the buffer capacity of the color-changing system. Accordingly, persons of skill in the art will readily recognize that appropriate adjustments of the buffer system may be required, and may make those adjustments. When used, the preservatives should generally be present in an amount up to about 1% by weight, with about 0.05% to about 0.5% being desirable.

Suitable organoleptic consistency modifiers or bioadhesives, include suspending agents, gelling agents or thickening agents such as cellulosics like methylcellulose, carrageenans like alginic acid and derivatives thereof, xanthan gums, gelatin, acacias and microcrystalline cellulose. Such organoleptic consistency modifiers may be used herein in an amount up to about 20% by weight, with about 1% to about 15% being desirable.

The following examples are provided for illustrative purposes only.

Example 1

A mouthwash product containing a color-changing system in accordance with this invention was prepared by placing into a vessel about 50 mls of a mouthwash base containing water, glycerin, poloxamer 335, PEG 600,

sodium lauryl sulfate, mint flavors and a benzoic acid/benzoate buffer system, which was prepared by combining about 0.025% (w/v) benzoic acid with about 0.018% (w/v) sodium benzoate.

Red cabbage extract (No. 3815, commercially available from Warner-Jenkinson) was added to the mouthwash base at room temperature with stirring in an amount of about 1.4 g/l [0.14% (w/v)]. The final pH of the sample was adjusted to about 4.0 with about 0.1 N HCl. The color of this color-changing system-containing mouthwash sample was observed to be red.

Thereafter, the pH value of this sample was increased to about 6.8 through the addition with stirring of about 0.01 N NaOH. The completion color of the sample was observed to be blue.

Example 2

A mouthwash product containing a color-changing system in accordance with this invention was prepared by placing into a vessel about 50 mls of the mouthwash base described in Example 1, supra.

Carmine liquid color (No. 3450, commercially available from Warner-Jenkinson) was added to the mouthwash base at room temperature with stirring in an amount of about 6.0 ml/l [0.6% (v/v)]. The final pH of the sample was adjusted to about 4.0 with about 0.1 N HCl. The color of this color-changing system-containing mouthwash sample was observed to be red.

Thereafter, the pH value of the mouthwash sample was increased to about 6.8 through the addition with stirring of 0.01 N NaOH. The completion color of the sample was observed to be purple-blue.

Example 3

A mouthwash product containing a color-changing system in accordance with this invention was prepared by placing into a vessel about 50 mls of a mouthwash base containing water, sorbitol solution, U.S.P. alcohol, poloxamer 407, sodium saccharine, sodium citrate, citric acid and benzoic acid.

Red cabbage extract (No. 3815) was added to a mouthwash base in an amount of about 0.14 grams. The final pH was adjusted to 4.0 with 0.1 N NaOH. The color of the mouthwash sample was observed to be red.

A subject then rinsed with about 30 mls of the mouthwash sample for a period of time of about 15 seconds, and then expectorated the used sample from the oral cavity. The expectorate was observed to be red.

After a period of time of about 1 hour was allowed to elapse from this rinsing, the same subject then rinsed with about 30 mls of the same sample for a period of time of about 30 seconds. This one hour waiting period was chosen to insure no residual effects from the first rinsing. The expectorate from this second rinsing was observed to be blue.

The lack of color change of the expectorate in the first rinsing and the color change of the expectorate observed in the second rinsing is indicative of how the present invention aids the user in complying with the manufacture's suggestions for recommended use.

While these examples have been presented for illustrative purposes, it is to be understood that variations and equivalents of these oral hygiene products containing a color-changing system exist both as to their formulations and as to their form of

delivery vehicle. And these variations and equivalents will provide suitable, if not comparable results, when viewed in connection with the results obtained from the above examples. Accordingly, such variations and equivalents are also intended to be encompassed by the claims which follow hereinafter.

WHAT IS CLAIMED IS:

1. An oral hygiene composition which changes color in the oral cavity comprising:

(a) an effective amount of one or more active oral hygiene ingredients;

(b) a color-changing system comprising

(i) a color-changing material capable of indicating changes in pH, and

(ii) a buffer system; and

(c) a vehicle suitable for delivering said color-changing system and said oral hygiene ingredients into the oral cavity.

2. The oral hygiene composition according to Claim 1, wherein the color-changing material is selected from the group consisting of tumeric, carminic acid and derivatives thereof, red cabbage extract, anthocyanin-containing materials, anthocyanin derivatives, bromocresol green, methyl red, bromocresol purple, chlorophenol red and combinations thereof.

3. The oral hygiene composition according to Claim 1, wherein the color-changing material is selected from the group consisting of bromothymol blue, phenol red, neutral red, cresol red, α-naphthophthalene, cresol purple and combinations thereof.

4. The oral hygiene composition according to Claim 1, wherein the color-changing material is carminic acid.

5. The oral hygiene composition according to Claim 1, wherein the color-changing material is red cabbage extract.

6. The oral hygiene composition according to Claim 2, wherein said buffer system is selected from the group consisting of benzoic acid/benzoate, citric

acid/citrate, acetic acid/acetate, succinic acid/succinate, phosphoric acid/phosphate and combinations thereof.

7. The oral hygiene composition according to Claim 3, wherein said buffer system is selected from the group consisting of imidazole·HCl, N-2-hydroxyethyl-piperazine-N'-2-ethanesulfonic acid, phosphoric acid/phosphate and combinations thereof.

8. The oral hygiene composition according to Claim 1, further comprising a member selected from the group consisting of orally-acceptable solvents, surfactants, sweeteners, flavoring agents, anti-foaming agents, humectants, lubricants, disintegrating agents, dyes, preservatives, organoleptic consistency modifiers and combinations thereof.

9. The oral hygiene composition according to Claim 1, wherein the active oral hygiene ingredients are selected from the group consisting of plaque-loosening ingredients, plaque removing ingredients, plaque-retarding ingredients, fluoride-releasing ingredients, anti-gingivitis ingredients, breath-freshening ingredients, bacteriocidal and bacteriostatic agents, desensitizing ingredients, tartar-control agents and combinations thereof.

10. The oral hygiene composition according to Claim 9, wherein the plaque-loosening ingredients are selected from the group consisting of dicalcium phosphates, silicon dioxide, calcium carbonate, aluminosilicates, sodium bicarbonate and combinations thereof.

11. The oral hygiene composition according to Claim 9, wherein the plaque-retarding ingredients are selected from the group consisting of the combination of thymol,

menthol, eucalyptol and methyl salicylate, quaternary ammonium compounds, triclosan and combinations thereof.

12. The oral hygiene composition according to Claim 11, wherein the quaternary ammonium compounds are selected from the group consisting of cetyl pyridinium chloride, benzethonium chloride, benzalkonium chloride and combinations thereof.

13. The oral hygiene composition according to Claim 9, wherein the anti-gingivitis ingredients selected from the group consisting of the combination of thymol, menthol, eucalyptol and methyl salicylate, bisbiguanides and combinations thereof.

14. The oral hygiene composition according to Claim 9, wherein the breath-freshening ingredients are selected from the group consisting of zinc salts, copper salts and combinations thereof.

15. The oral hygiene composition according to Claim 9, wherein the bacteriocidal and bacteriostatic agents are selected from the group consisting of the combination of thymol, menthol, eucalyptol and methyl salicylate, quaternary ammonium compounds, bisbiguanides, triclosan and combinations thereof.

16. The oral hygiene composition according to Claim 9, wherein the desensitizing ingredients are selected from the group consisting of stannous fluoride, citric acid and salts thereof, potassium nitrate, strontium chloride and combinations thereof.

17. The oral hygiene composition according to Claim 9, wherein the tartar-control agents are selected from the group consisting of stannous fluoride, pyrophosphates, chelating agents and combinations thereof.

18. The oral hygiene composition according to Claim 1, wherein said vehicle is selected from the group consisting of liquids, suspensions, pastes, gels, balms, ointments, salves, creams, chewing gums, lozenges, tablets and powders.

19. A dentifrice capable of changing color after being retained with the oral cavity for a duration sufficient to assure an effective oral hygiene treatment to the oral cavity comprising:

(a) an effective amount of one or more active oral hygiene ingredients;

(b) a color-changing system comprising

(i) a color-changing material capable of indicating changes in pH, and

(ii) a buffer system; and

(c) a vehicle suitable for delivering said color-changing system and said oral hygiene ingredients into the oral cavity.

20. A mouthwash capable of changing color after being retained within the oral cavity for a duration sufficient to assure an effective amount of an oral hygiene treatment to the oral cavity comprising

(a) an effective amount of one or more active oral hygiene ingredients;

(b) a color-changing system comprising

(i) a color-changing material capable of indicating changes in pH, and

(ii) a buffer system; and

(c) a vehicle suitable for delivering said color-changing system and said oral hygiene ingredients into the oral cavity.

21. A chewing gum capable of changing color after being retained within the oral cavity for a duration sufficient to assure an effective amount of an oral hygiene treatment to the oral cavity comprising:

(a) an effective amount of one or more active oral hygiene ingredients,

(b) a color-changing system comprising

(i) a color-changing material capable of indicating changes in pH, and

(ii) a buffer system; and

(c) a vehicle suitable for delivering said color-changing system and said oral hygiene ingredients into the oral cavity.

22. A method of using an oral hygiene composition to assure an effective oral hygiene treatment to the oral cavity, said method comprising the steps of:

(a) delivering into an oral cavity an oral hygiene composition comprising:

(i) an effective amount of one or more active oral hygiene ingredients,

(ii) a color-changing system, and

(iii) a vehicle suitable for delivering said color-changing system and said oral hygiene ingredients into the oral cavity;

(b) retaining the oral hygiene composition within the oral cavity for a period of time sufficient to provide an effective oral hygiene treatment, said period of time being determined when said color-changing material changes color from an initial color to a completion color indicating that an effective oral hygiene treatment has been provided; and

(c) expectorating the used oral hygiene composition from the oral cavity.

INTERNATIONAL SEARCH REPORT

Ir tional Application No
PCT/US 95/15372

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A61K7/16 A23G3/30

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61K A23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO,A,92 04007 (BEECHAM INC.) 19 March 1992 cited in the application see examples 2-4	1,20
X	US,A,4 150 106 (J.ASSAL) 17 April 1979 see claims 1-7	1,2,6,8, 9,18,19, 22
X	US,A,4 568 534 (R.STIER) 4 February 1986 see column 1, line 57 - column 2, line 5 see column 2, line 16-58; claims 1-8	1,8-10, 18,19,22

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
4 March 1996	12. 03. 96

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INTERNATIONAL SEARCH REPORT

	tional Application No
	PCT/US 95/15372

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Y		1-3,19, 21,22

Y	FR,A,2 151 780 (CONCENTRADOS NACIONALS S.A.) 20 April 1973 see claim 1	21

Y	PATENT ABSTRACTS OF JAPAN vol. 9 no. 205 (C-299) [1928] & JP,A,60 075409 (TAIHEI KOGYO K.K.) 27 April 1985, see abstract	1-3,19, 22

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